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Use of FTIR Analysis to Control the Self-Healing Functionality of Epoxy Resins

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1. Introduction

In recent years polymer composites are increasingly used to replace the traditional metal alloys in structural applications, ranging from civil infrastructure to high performance vehicles such as racing cars and military aircraft. This popularity is due to their lower weight, as well as to a continuous improvement of their performance aided in recent years by nanotechnology. However, limited storage stability and reliability are critical for polymer composites designed for structural applications [1-4]. In fact, in service, they are subject to damage due to microcracks that are produced in the structure under the action of various kinds of stresses, for example: a) mechanical vibrations or various types of mechanical stresses, b) sudden temperature changes, c) irradiation by electromagnetic radiation causing direct or indirect rupture of chemical bonds (UV light, γ rays, etc.), d) intentional or inadvertent contact with chemical substances that adversely affect the structure, e) various factors which in combination can contribute to compromising the integrity of the structure. Internal damage is difficult to detect and, once developed, even more difficult to repair. This critical point is a real problem in the field of aeronautic vehicles. In fact, for large components, such as parts of primary structures, several non-destructive damage detection techniques have been developed including ultrasonic, infrared thermography, x-ray tomography, and computerized vibro-thermography. This technology has helped to detect damage but repair of this damage has been limited to reinforced patch bonding and/or bolting. Actually, durability and reliability are still problematic in the field of these structural materials; in fact, in order to achieve the mechanical strength required for many structural applications, highly cross-linked polymeric materials are necessary. The trade off for this gain in mechanical strength is that the resulting materials tend to be brittle and are therefore more prone to developing cracks through normal usage, ultimately failing.

In addition to conventional methods for damage detection, and common repair methods, there has recently been the development of self-healing composites which are expected to significantly extend the life of polymeric components by autonomically healing micro-cracks

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whenever and wherever they develop [5-15]. For structural applications, self-healing systems are of great interest because they would allow to overcome not only some difficulties connected to damage diagnosis, but also the following appropriate interventions to restore the material functionality. Airlines for example see polymeric composites' potential to cut fuel costs and save on maintenance very attractive, therefore, are willing to entertain the idea to put into action self-healing composites in the development of different materials for aeronautical applications. The challenge facing materials scientists is to assure these systems must be able to stem fatigue damage and preserve their integrity, increase their life span, reduce maintenance costs and provide safety during use. A lot of strategies were formulated up to now in the development of self-healing materials [5,16-27].

One of the strategies to manufacture a thermosetting self-repair material seems to be the storage of healing agents inside composites that restore the strength of the materials after damage.

The first demonstration of self-healing in an engineered material, an epoxy matrix, occurred in 2001 [5] by a team from the University of Illinois (USA). This self-healing system for thermosetting materials is very interesting also in terms of design. The concept was based on the introduction of a microencapsulated healing agent and suspended catalyst phase in a polymer matrix. Since that time, advancement has been made in the field following this conceptual approach, while at the same time alternative concepts have emerged in the scientific literature. Microencapsulation and thermally reversible networks [28] are the two main strategies used for the development of self-healing thermosetting materials. Microencapsulation is the first and most studied self-healing concept. The initial self-healing epoxy system developed by White et al. involves the incorporation of a microencapsulated healing agent, dicyclopentadiene (DCPD) and a dispersed solid catalyst, bis(tricyclohexylphosphine) benzyldiene ruthenium (IV) dichloride (called Grubbs' catalyst) in an epoxy-amine network. In these systems, an approaching crack ruptures embedded microcapsules releasing a polymerizer agent into the crack plane through capillary action. Polymerization of the healing agent is triggered by contact with the embedded catalyst, bonding the crack faces. In these systems the efficiency of self-repair function, in terms of trigger, speed and yield, is related to ring-opening metathesis polymerization of the healing agent by appropriate catalysts. The healing agent is a microencapsulated liquid monomer that must include a long shelf life, prompt deliverability, high reactivity, and low volume shrinkage upon polymerization [29]. The monomer most often used as the healing agent for the manufacture of these first ingenious systems is dicyclopentadiene (DCPD) [5,29]. Very recently, however, blends of DCPD/5-ethylidene-2-norbornene (DCPD/ENB) or DCPD/ 5-norbornene-2carboxylic acid have also been proposed [30]. Thermosetting auto-repair polymers, which have been proposed so far, include Grubbs' first-generation catalyst (G1); [5,17,29-30] and currently, the possibility of applying other ruthenium catalysts for ring-opening metathesis polymerization-based self-healing applications is being evaluated. This system is a challenge for epoxy structural composites: however, some drawbacks have to be re-evaluated in order to be fully applied to advanced applications. These mainly regard the thermal stability of the Grubbs' catalyst inside the epoxy resin during the curing cycle and the impossibility to utilize primary amines as hardeners, since they can poison the catalyst.

This last drawback forces the use of compounds which have not been fully explored in literature as hardener agents, especially with regard to the cure behavior and mechanical

properties of the manufactured materials. Since 2003, a large number of parameters were investigated by the team from the University of Illinois and other research groups. In a recent paper, the dissolution properties, initial polymerization kinetics, chemical stabilities, and thermal stabilities were analyzed for three catalysts: Grubbs' first (G1) and second generation (G2) catalysts and Hoveyda-Grubbs' second generation catalyst (HG2) [30]. Ruthenium-based catalysts are reported as exhibiting great functional group tolerance, as well as greatly enhanced air and water stability [30,31]. However, thermolytic decomposition can limit the usefulness of these ruthenium systems in self-healing composites based on epoxy resins [32-33]. This is a crucial aspect for self-healing systems in aeronautic applications because, even if linked to the cost reduction from a process aspect, resins have been developed with low temperature manufacturing (under 100 °C), yet the problem of the material treatment at high temperature is not resolved.

In fact, we generally need a glass transition temperature after wet aging of 110 °C minimum, and a curing temperature equal to or less than 100 °C is not enough. To achieve this goal it is necessary to make a post cure with a temperature that could be as high as 180 °C. For this reason, we have carried out our investigation in the temperature range of 150-180 °C. It is worth noting that there are critical issues in the use of epoxy precursors in conjunction with Grubbs metathesis catalysts, because in self healing composites based on epoxy resins, ruthenium systems give rise to a reaction with the oxirane rings of the epoxy precursors, and, therefore, the metathesis catalyst was not subsequently able to promote the polymerization of the reactive monomer, thus losing the self-healing ability. Such phenomena could strongly limit the use in the practice, in self-healing composite materials, of Grubbs' catalysts. In particular, in this chapter, we show that selecting the appropriate curing cycle as well as the specific chemical formulation, the catalyst remains intact in the formed epoxy matrix during the curing process, and is thus capable of subsequently performing its catalytic activity of the polymerization of the reactive monomer consisting in the cyclic olefin (5-ethylidene-2-norbornene -ENB-), when the latter comes out of a microcapsule affected by a crack. Studies on the choice of an appropriate curing cycle for a different self-healing formulation have already been reported in previous papers [32-35]. Another drawback to overcome in the employment of the ROMP catalysts is related to the local availability of the catalyst particles. In particular, the catalyst for the metathesis reaction is embedded in the precursors of the epoxy matrix in the form of solid particles, i.e. powders with different morphology and crystallographic modifications [30]. In practice, the effective concentration of the catalyst depends on the availability of the aforesaid particles at the level of the fracture and on the rate of dissolution of the catalyst in the reactive monomer (healing agent) within the polymer matrix. Even with high concentrations of catalyst particles exposed at the level of the fracture, the effective concentration of the catalyst could be relatively low because of limited rates of dissolution of the catalyst. The rate of dissolution of the catalyst depends not only on the chemical nature of the various components, but also on morphological and structural characteristics of the catalyst particles. It has been found in practice that the presence of the catalyst in the form of crystalline powders has some critical aspects relating to the uniform availability of the catalyst in all the zones in which a micro-crack can potentially develop, compromising the effectiveness of the self-healing process [35].

Figure 1 illustrates as, independently from the catalyst amount, large crystals of catalyst particles can cause loss of catalytic activity in many zones. In addition, a very effective self-

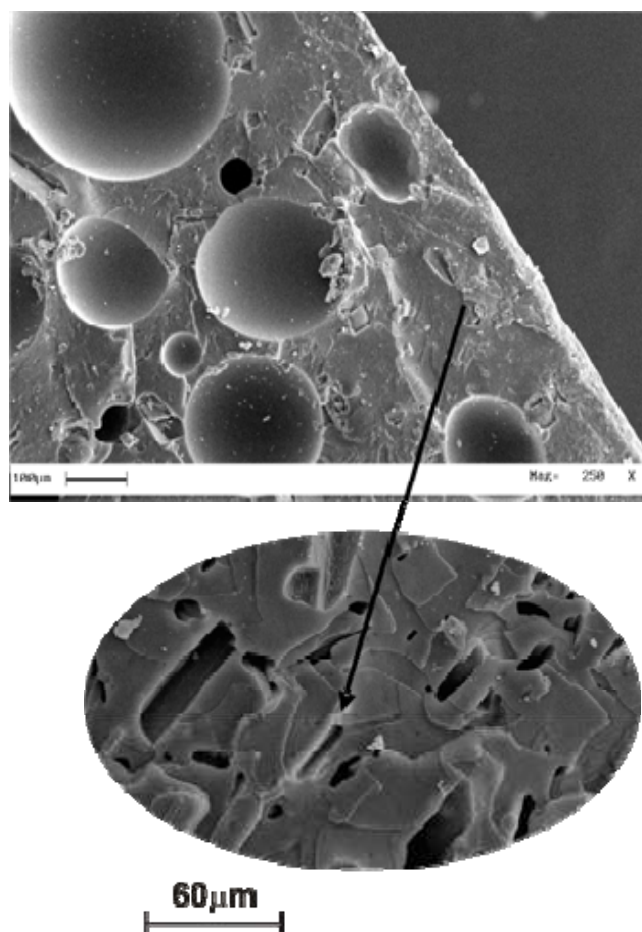


Fig. 1. SEM micrograph of the section surface of the self-healing epoxy specimen in which we can observe the empty geometrical places left by the catalyst in the epoxy matrix (see arrow)

healing system also needs the study and control of the capsule dimensions that act as reserve of healing agent.

Another aim of this chapter is therefore to study a process for the development of a self-healing composite, which does not have the drawbacks mentioned above with respect to the catalyst morphology. This purpose is achieved by a process for the manufacture of a self-healing composite material comprising a preliminary step of dispersing at molecular level the catalyst in an epoxy mixture containing healing agent in nanometric vessels. This solution allows to obtain the catalyst dispersed at molecular level and also able to react with nano-encapsulated healing agent.

2. Materials

The *epoxy matrix composite* was prepared by mixing an epoxy (Bisphenol A diglycidyl ether - Acronym BADGE) with a reactive diluent (1,4-Butanediol-diglycidyl ether- Acronym BDDGE) which was used in small percentage to reduce the viscosity of the material, to improve handling and ease of processing and to optimize consequently performance properties. These resins, both containing an epoxy, were obtained by Sigma-Aldrich. The *curing agent* investigated for this study is an anionic initiator Phenol, 2,4,6-

tris[(dimethylamino) methyl] (Trade name Ancamine K54). This hardener agent was already used for self-healing formulations [5]. The catalyst, Hoveyda-Grubbs' first generation (Dichloro(o-isopropoxyphenylmethylene)(tricyclo-hexylphosphine)ruthenium(II) (catalyst HG1) also obtained from Aldrich was used to manufacture the epoxy matrix. It was dispersed at molecular level into the epoxy matrix. The healing agents used in this work were dicyclopentadiene (DCPD), which was obtained from Acros Organics, and 5-ethylidene-2-norbornene (ENB), which was obtained from Sigma-Aldrich. Figure 2 shows the chemical structures of compounds used in this work.

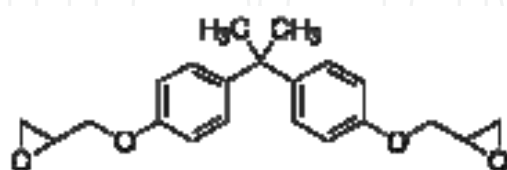
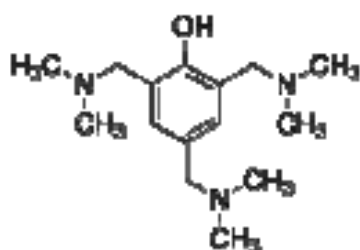
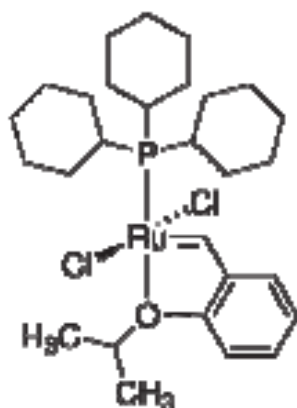
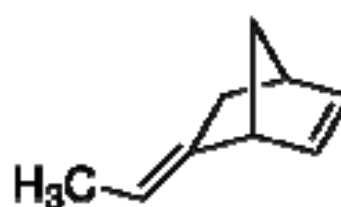
**BADGE****ANKAMINE K 54****BDDGE****HG1 CATALYST****DCPD****ENB**

Fig. 2. Chemical structures of compounds used for the self-healing system.

2.1 Epoxy specimen manufacture

2.1.1 Epoxy matrix

Sample EBA was obtained by mixing BADGE with BDDGE diluent at a concentration of 90%: 10% (by wt) epoxide to diluent. Ancamine K54 was added at a concentration of 10:100 (by wt) hardener to mixture (BADGE and BDDGE).

2.1.2 Self-healing epoxy system

Self-healing epoxy system was obtained by dispersing ENB/DCPD(5%)-filled nanocapsules at a concentration of 20 wt% into the epoxy matrix.

Healing efficiency was also measured by carefully controlled fracture experiments for both the virgin and the healed materials using a well established protocol [5].

2.1.3 Microcapsule manufacture

The microcapsules, with the outer shell composed of poly(urea-formaldehyde) and the inner shell of ethylene maleic anhydride copolymer (EMA) were prepared by *in situ* polymerization in an oil-in-water emulsion in accord with a procedure already described in previous papers [5, 33]. The only change, with respect to the aforementioned synthesis procedure, consisted of using as healing agent a blend of 5ethylidene-2norbornene (95 wt%) and DCPD (5 wt%). According to such a procedure, a desired dimension range can be selected by a suitable variation of the process parameters during the synthesis stage, and/or with the use of molecular sieves. The capsules used to manufacture the self-healing system are shown in Fig. 3. The analysis of the capsule size distribution (obtained from a series of 20

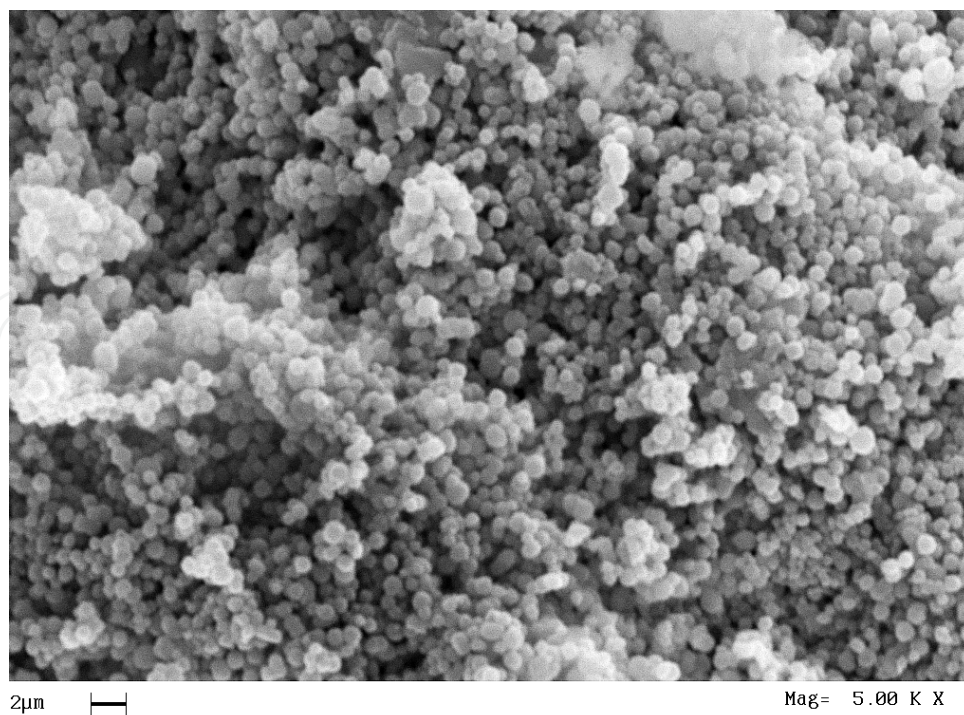


Fig. 3. Scanning electron microscope image of micro and nanocapsules at magnification 5.00 K x

samples) show that the largest part are characterized by a spherical form with average diameter of about 500-600 nm.

The efficiency of the microencapsulation process for the above described formulation was not evaluated because for this new formulation other experiments on the self-healing efficiency were carried out. This analysis does not require a hard-working procedure if we use the infrared spectroscopy; by way of example, hereafter, in the section “Methodologies” we report a procedure to analyze the efficiency of the microencapsulation process for microcapsules only filled with DCPD.

3. Methodologies

3.1 Efficiency of the microencapsulation process

FTIR analysis can be performed to control the success of the microencapsulation process. For this investigation, all the microcapsule fractions were powdered in a mortar; a first small fraction was dried under vacuum and analyzed by FT/IR spectroscopy (red spectrum) (see Figure 4), while another fraction was directly treated with Grubbs’ catalyst powder and again analyzed by FT/IR (brown spectrum). Mixing and powdered the fraction of

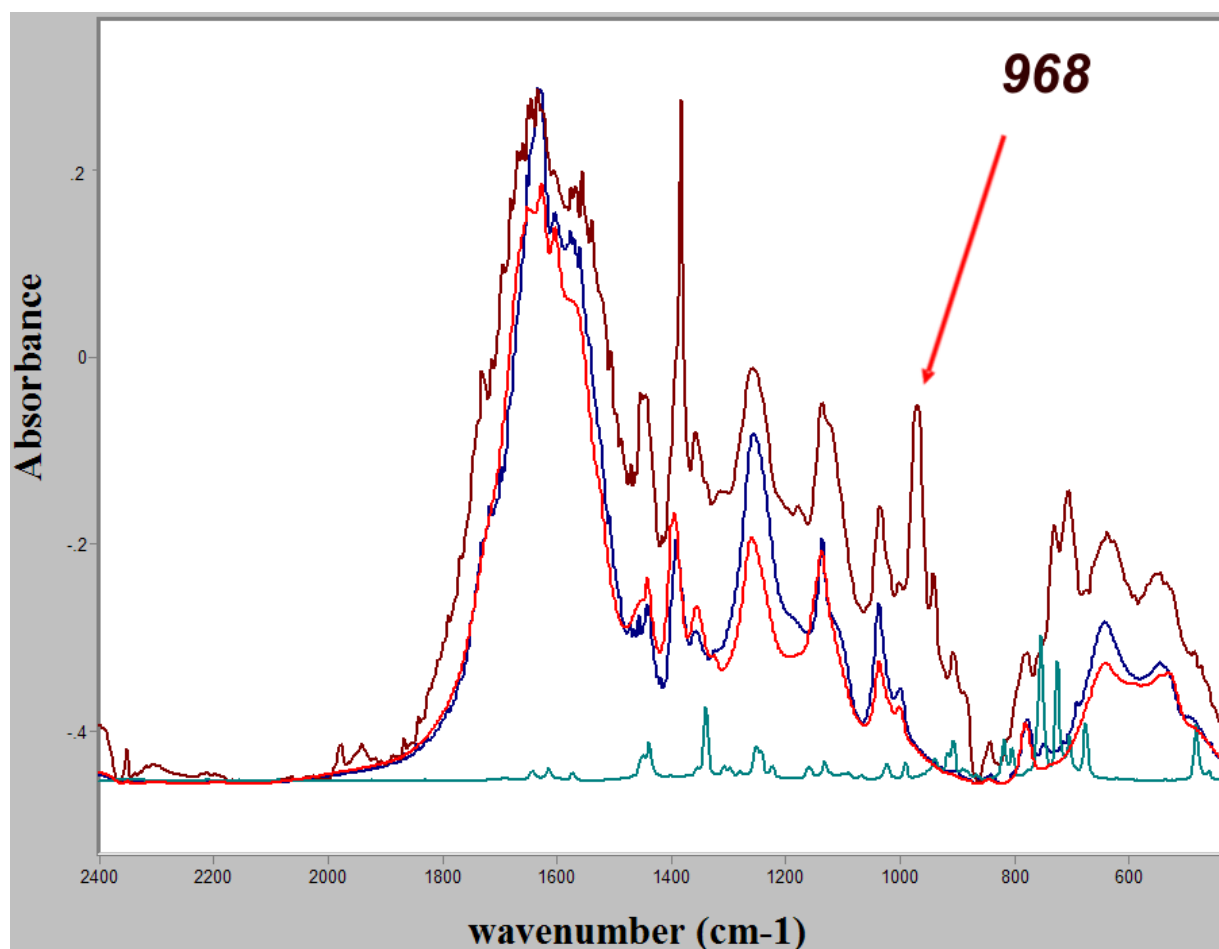


Fig. 4. FTIR spectra of microcapsules (blue spectrum), DCPD (green spectrum), of the microcapsules fraction dried under vacuum (red spectrum) and microcapsules fraction treated with Grubbs’ catalyst powder (brown spectrum).

microcapsules (containing DCPD) with the Grubbs catalyst instantaneously initiates ROMP at room temperature. ROMP polymers can display a very rich microstructure. Depending on the monomer, different characteristics can be observed, among these cis/trans isomerism, tacticity, etc.. Cis/trans isomerism is present in all ROMP polymers and relatively easy to quantify using spectroscopic techniques. In fact, in the brown spectrum we can observe the strong and sharp signal at 968 cm^{-1} due to the carbon hydrogen bending vibration of a trans carbon double bond. This signal was already assigned to the absorption of trans poly(DCPD) fractions^{5,36}. Using this procedure ROMP of DCPD primarily produces trans double bonds as observed for the ROMP of DCPD by Grubbs first generation catalyst. In figure 4 FT/IR spectra of DCPD and microcapsules, as obtained from the synthesis, were also reported for comparison. In particular, in the analyzed spectral range, a comparison between red and brown spectra shows many common signals due to poly(urea-formaldehyde) of the microcapsule wall. We can observe, in fact, some of the vibrational mode for the CH_2 group ($\delta_s\text{CH}_2$ at 1465 cm^{-1} – in-plane bending or scissoring; ωCH_2 and τCH_2 between $1350\text{--}1150\text{ cm}^{-1}$ – out-of-plane bending or wagging and/or twisting; ρCH_2 at 720 cm^{-1} – in-plane bending or rocking) and the carbonyl frequency (C=O) of the wall tertiary amides. This absorption occurs in the spectral range of $1680\text{--}1630\text{ cm}^{-1}$ as expected (the C=O absorption of amides occurs at lower frequencies than “normal” carbonyl absorption due to the resonance effect. The resonance effect increases the C=O bond length and reduces the frequency of absorption³⁷).

The highlighted peak at 968 cm^{-1} characteristic of ring-opened poly(DCPD) is present only in the brown spectrum. It clearly evidences that the embedded DCPD is active in the metathesis reaction. As shown by the FT/IR analysis reported in figure 4, the amount of DCPD encapsulated is a sufficient quantity to activate the ROMP reaction [32 – see Supp. Inf.].

3.2 Evaluation of the catalytic activity

In the development of our self-healing epoxy resins, the evaluation of the catalytic activity was investigated for the epoxy matrix (formulation without nanocapsules).

The procedure adopted for preparing this mixture is as follows. The epoxy precursor (BADGE) was mixed mechanically with the reactive diluent (BDDGE) at a temperature of 90°C , maintained with an oil bath, and then the catalyst HG1 in the form of crystalline powder was added. The catalyst was dispersed at molecular level by mechanical agitation of the mixture maintained at 90°C for 90 minutes. To verify complete dispersion and dissolution of the catalyst, and that its catalytic activity remained unchanged, spectroscopic investigation was carried out. For this purpose, four drops of the mixture were deposited on a slide for light microscopy. Complete transparency, which is achieved when the catalyst is completely dissolved, can be verified by light microscopy with observation in transmission. Two drops of ENB were added to the aforesaid drops of mixtures. A thin solid film of metathesis product, whose FT/IR spectrum is shown in Fig. 5, formed immediately.

This spectrum shows a peak at 966 cm^{-1} , which is an indication of the formation of the metathesis product and hence of the fact that the activity of the catalyst has not been compromised by the chemical nature of the oligomers, by the temperature and by the treatments of mechanical mixing.

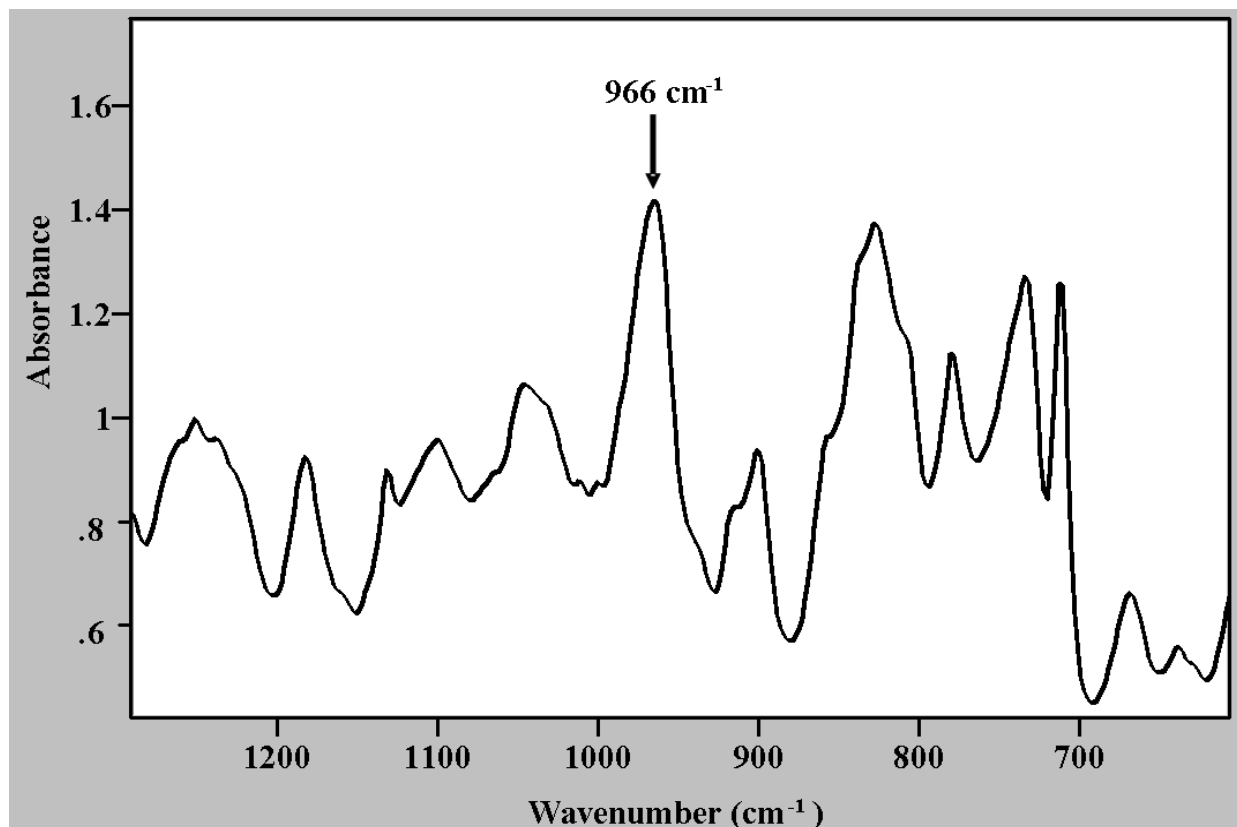


Fig. 5. FT/IR spectrum of the solid film (metathesis product)

The transparent mixture containing the completely dissolved catalyst was then taken out of the oil bath and left to cool 50°C, and then the curing agent was added to it. The mixture thus obtained was cured in a two-stage process. The first stage was carried out at a temperature of 80°C for 3 hours, while the second stage was carried out according to three variants, i.e. at the three different temperatures of 125, 150 and 170°C.

Figs. 6a, 6b, 6c and 6d show FT/IR spectra of the cured material respectively after the intermediate stages, and the second stage carried out with the three variants stated above, to which ENB was then added in the same way as was done after the preliminary step of dissolution of the catalyst.

We can observe that, in conjunction with different signals of the epoxy precursors (among which the C-O-C stretch at 1247 cm⁻¹ and the symmetric stretch at 1039 cm⁻¹) all the spectra show the band at 966 cm⁻¹.

The presence in all cases of the peak at 966 cm⁻¹, indicating formation of the metathesis product, proves that the catalytic activity of the HG1 catalyst within the epoxy matrix remained unchanged after the described treatments. It can be seen, in particular, from the FT/IR spectra that, after the treatment at 110°C, the mixture can be cured up to 170°C for 2 hours without compromising the catalytic activity. This high temperature increases the cross-linking degree, as we can deduce observing the signal at 916 cm⁻¹ in the spectra of figs. 6a-6d. The very small intensity of the signal at 916 cm⁻¹ in the spectrum of fig. 6a and the further progressive decrease up to the disappearance in the spectrum of fig. 6d indicates that almost the epoxy rings have reacted for a treatment up to 110 °C (the signals at 916 cm⁻¹

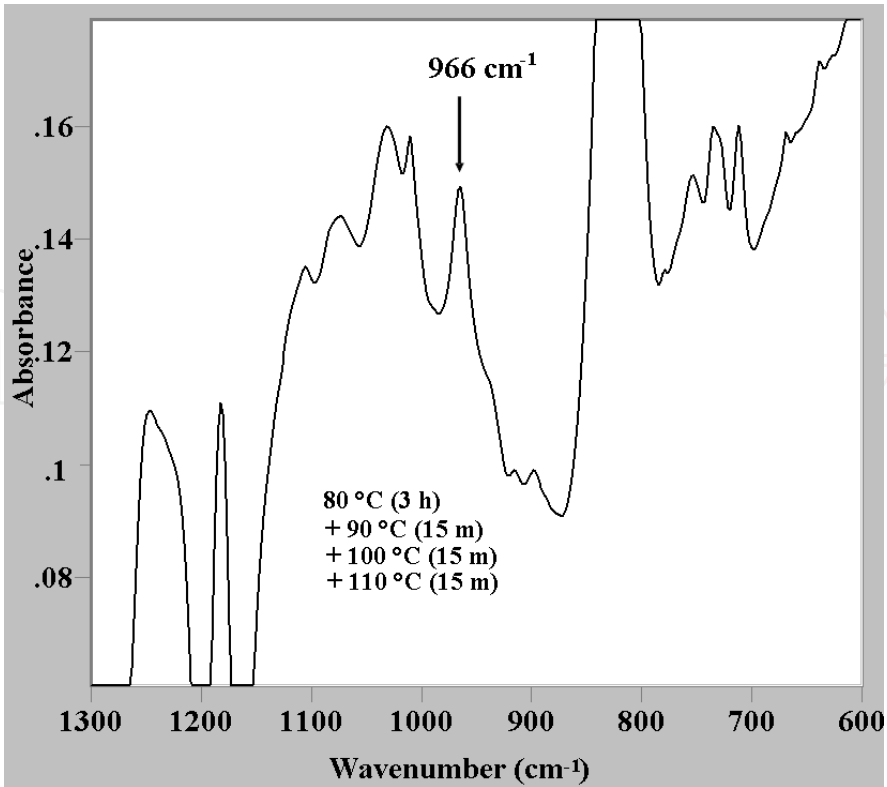


Fig. 6a. FT/IR spectrum of the solid film (metathesis product) in the material cured up to 110 °C.

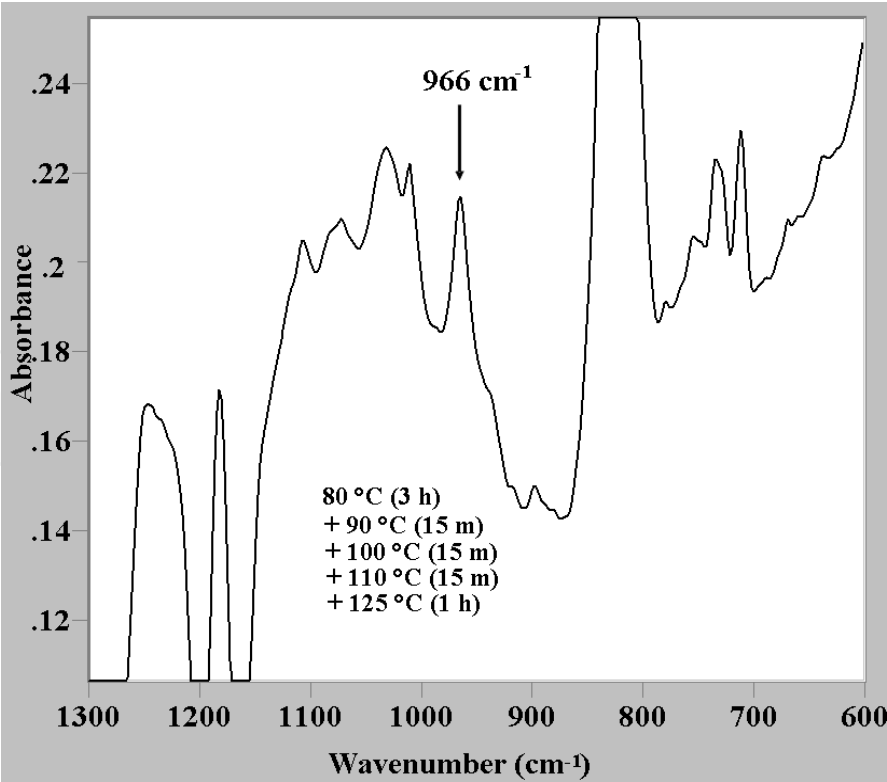


Fig. 6b. FT/IR spectrum of the solid film (metathesis product) in the material cured up to 125 °C

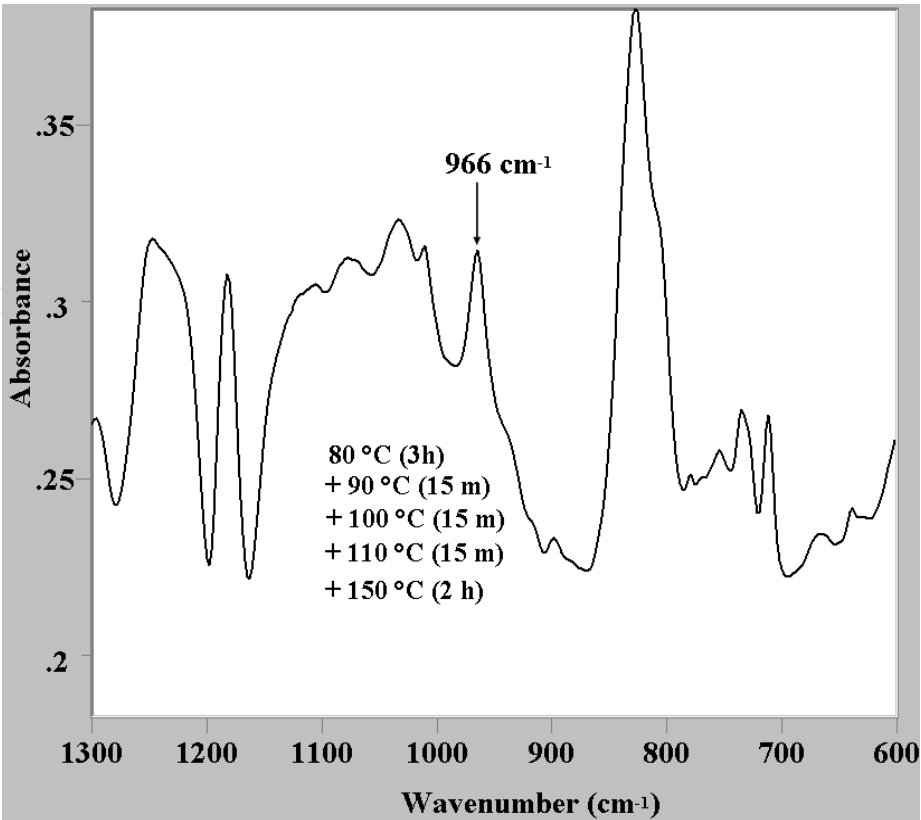


Fig. 6c. FT/IR spectrum of the solid film (metathesis product) in the material cured up to 150 °C

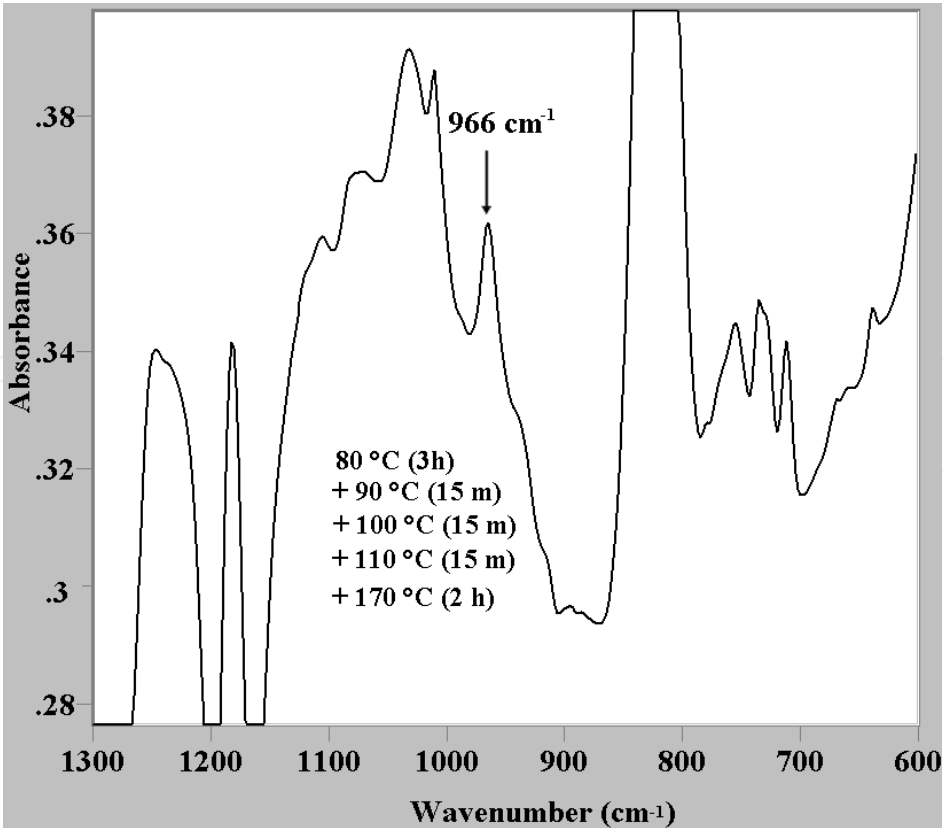


Fig. 6d. FT/IR spectrum of the solid film (metathesis product) in the material cured up to 170 °C

is characteristic of the epoxy groups and assigned to asymmetrical ring stretching in which the C-C bond is stretching during contraction of the C-O bond ³⁷).

Self-healing epoxy system was obtained by dispersing ENB/DCPD(5% wt%)-filled nanocapsules at a concentration of 20 wt% into the epoxy matrix using for the curing process the same procedure above described for the epoxy matrix. Use of a mixture of ENB with low concentrations of DCPD as healing agent was used because It can greatly increase the degree of crosslinking of the metathesis product especially at extremely low temperatures. For example, by carrying out the ring opening metathesis reaction of an ENB/DCPD mixture (at 5% of DCPD) in the molar ratio 1:1000 (Hoveyda-Grubbs 1 catalyst/monomer) at a temperature of -53°C, the degree of crosslinking is found to be 57% (with a degree of conversion of 84%) after a reaction time of 7 hours. In similar conditions, the degree of crosslinking of ENB on its own is 10% with a degree of conversion of 100% [35].

A simplified scheme of the self-healing formulation is shown in Figure 7.

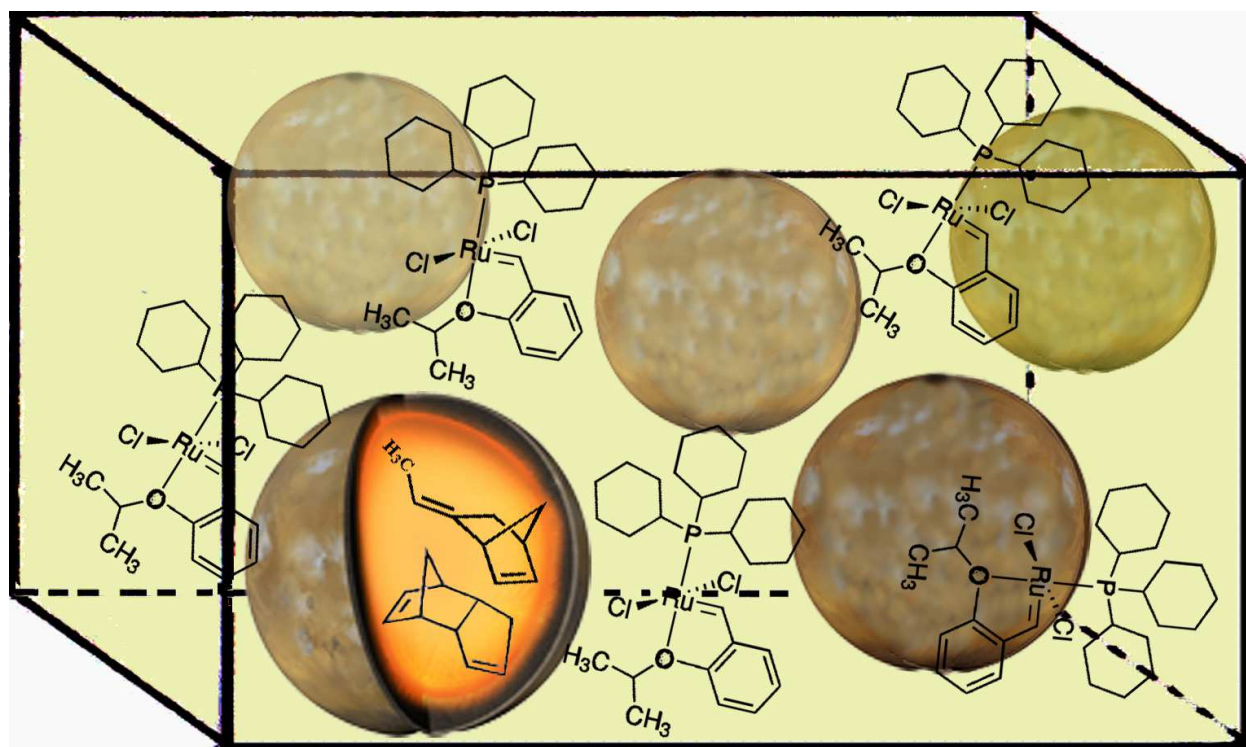


Fig. 7. Scheme of self-healing formulation which includes: a) capsules containing a polymerizer agent (DCPD and ENB); b) a catalyst for the polymerizer (HG1).

3.3 Healing efficiency of the self-healing sample

Crack healing efficiency, η , (defined as the ability to recover fracture toughness) was evaluated for the sample cured up to 170 °C using a tapered double-cantilever beam (TDCB) [23,38] geometry. The healing efficiency η , calculated as the ratio of critical fracture loads for the healed and virgin samples, is obtained from data shown in Figure 8 where we report the Load-Displacement curves for a sample with 5% of Hoveyda-Grubbs I Catalyst and 20% of microcapsules (ENB+DCPD) filled, cured up to 170 °C. The healing efficiency is 72 %.

Fig. 9 shows the sample geometry for getting quantitative results on the self-healing functionality. The figure also reports the morphology of an healed sample after a controlled damage.

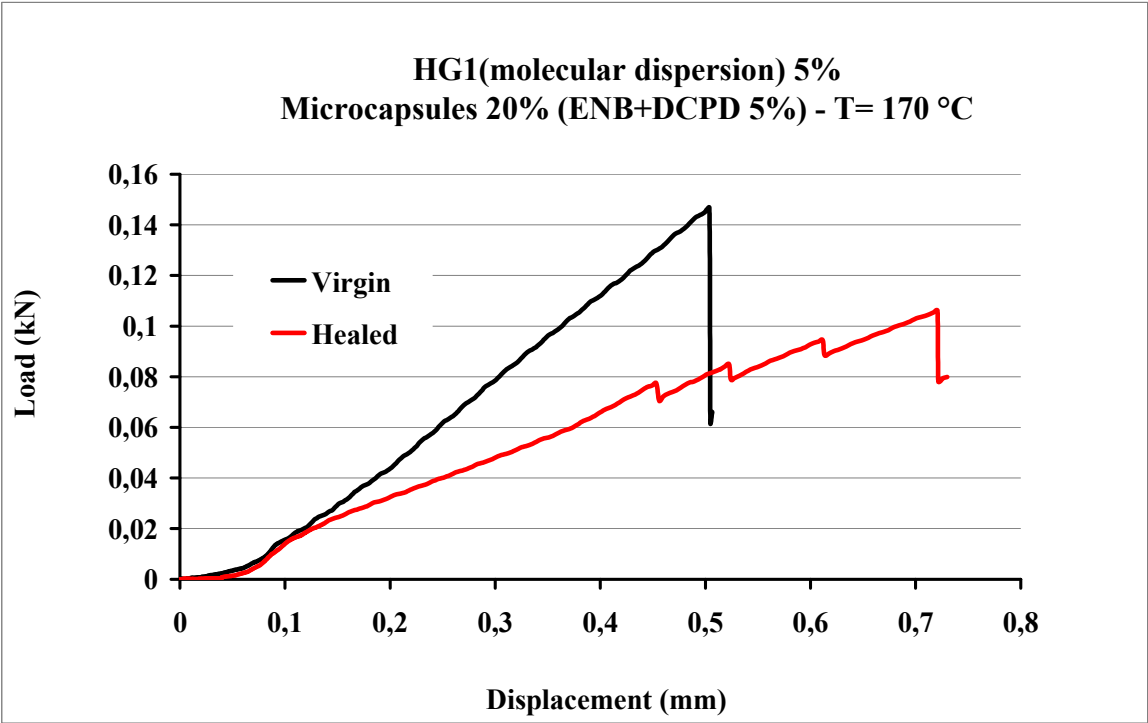


Fig. 8. Load-Displacement curves for Virgin (black curve) and Healed (red curve) sample.

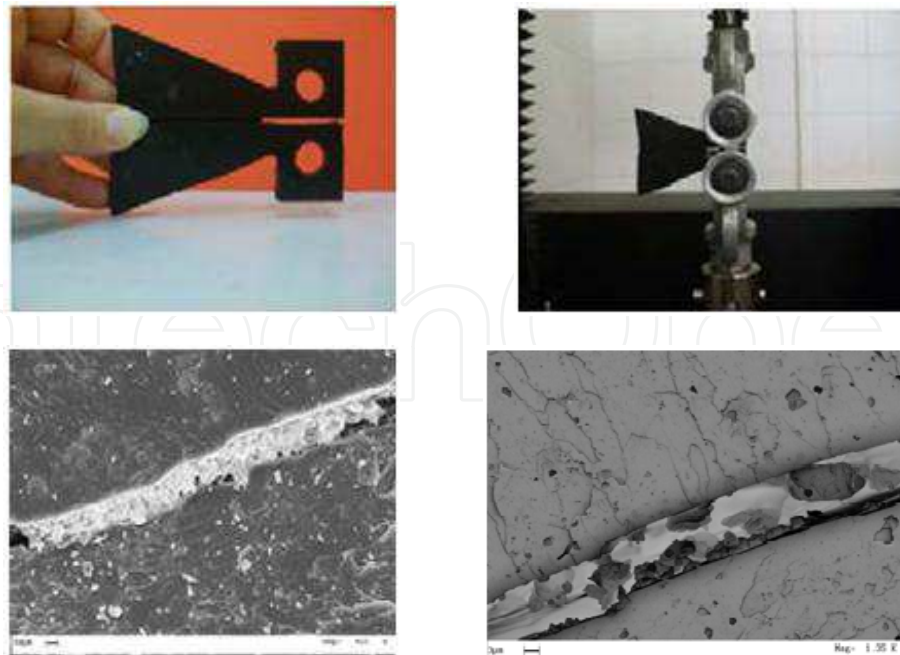


Fig. 9. Samples for getting quantitative results on the self-healing functionality (the first two images on the top); SEM images of healed crack faces closed by means of the metathesis product inside a crack after a (TDCB) test (images on the bottom).

4. Conclusions

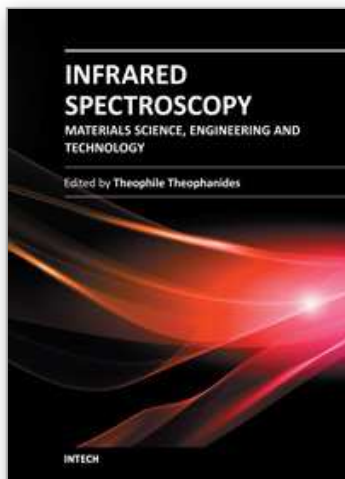
We have formulated, prepared and characterized a multifunctional autonomically healing composite with a self-healing efficiency higher than 70 %. Carrying out the curing process in several stages at increasing temperature makes it possible to avoid deactivation of the catalyst. In fact, choosing a relatively low temperature in the first stage means that only the curing agent, and not the catalyst, reacts with the oxirane rings of the epoxy precursor. Therefore, the catalyst remains intact in the epoxy matrix that has formed and is thus able subsequently to perform its catalytic action of polymerization of the reactive monomer, when the latter is released from the vessel and interacts with the epoxy matrix containing the catalyst. Infrared Spectroscopy proves to be a useful way to identify metathesis product directly inside the epoxy resin during the curing reactions of epoxy formulations containing catalyst powder dispersed at molecular level.

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